PATENT COOPERATION TREATY

PCT

INTERNATIONAL PRELIMINARY REPORT ON PATENTABILITY

(Chapter II of the Patent Cooperation Treaty)

(PCT Article 36 and Rule 70)

Applicant's or agent's file reference 034169.WO	**FOR FURTHER ACTION**	See Form PCT/IPEA/416
International application No. PCT/NL2004/000796	International filing date *(day/month/year)* 12.11.2004	Priority date *(day/month/year)* 25.11.2003
International Patent Classification (IPC) or national classification and IPC B09B3/00, B03B9/06, C22B43/00		
Applicant VERMEULEN, Anthonius Hendricus Maria		

1. This report is the international preliminary examination report, established by this International Preliminary Examining Authority under Article 35 and transmitted to the applicant according to Article 36.
2. This REPORT consists of a total of 6 sheets, including this cover sheet.
3. This report is also accompanied by ANNEXES, comprising:
   a. ☒ *sent to the applicant and to the International Bureau)* a total of 9 sheets, as follows:
      ☒ sheets of the description, claims and/or drawings which have been amended and are the basis of this report and/or sheets containing rectifications authorized by this Authority (see Rule 70.16 and Section 607 of the Administrative Instructions).
      ☐ sheets which supersede earlier sheets, but which this Authority considers contain an amendment that goes beyond the disclosure in the international application as filed, as indicated in item 4 of Box No. I and the Supplemental Box.
   b. ☐ *(sent to the International Bureau only)* a total of (indicate type and number of electronic carrier(s)) , containing a sequence listing and/or tables related thereto, in computer readable form only, as indicated in the Supplemental Box Relating to Sequence Listing (see Section 802 of the Administrative Instructions).

4. This report contains indications relating to the following items:

☒	Box No. I	Basis of the opinion
☐	Box No. II	Priority
☐	Box No. III	Non-establishment of opinion with regard to novelty, inventive step and industrial applicability
☐	Box No. IV	Lack of unity of invention
☒	Box No. V	Reasoned statement under Article 35(2) with regard to novelty, inventive step or industrial applicability; citations and explanations supporting such statement
☐	Box No. VI	Certain documents cited
☐	Box No. VII	Certain defects in the international application
☐	Box No. VIII	Certain observations on the international application

Date of submission of the demand	Date of completion of this report
08.06.2005	04.11.2005
Name and mailing address of the international preliminary examining authority: European Patent Office - P.B. 5818 Patentlaan 2 NL-2280 HV Rijswijk - Pays Bas Tel. +31 70 340 - 2040 Tx: 31 651 epo nl Fax: +31 70 340 - 3016	Authorized Officer van der Zee, W Telephone No. +31 70 340-2797

INTERNATIONAL PRELIMINARY REPORT ON PATENTABILITY

International application No.
PCT/NL2004/000796

Box No. I Basis of the report

1. With regard to the **language**, this report is based on the international application in the language in which it was filed, unless otherwise indicated under this item.

   ☐ This report is based on translations from the original language into the following language , which is the language of a translation furnished for the purposes of:
   - ☐ international search (under Rules 12.3 and 23.1(b))
   - ☐ publication of the international application (under Rule 12.4)
   - ☐ international preliminary examination (under Rules 55.2 and/or 55.3)

2. With regard to the **elements*** of the international application, this report is based on *(replacement sheets which have been furnished to the receiving Office in response to an invitation under Article 14 are referred to in this report as "originally filed" and are not annexed to this report)*:

Description, Pages	
6, 7	as originally filed
1-5, 5A	received on 19.08.2005 with letter of 18.08.2005
Claims, Numbers	
1-16	received on 19.08.2005 with letter of 18.08.2005
Drawings, Sheets	
1/1	as originally filed

   ☐ a sequence listing and/or any related table(s) - see Supplemental Box Relating to Sequence Listing

3. ☐ The amendments have resulted in the cancellation of:
   - ☐ the description, pages
   - ☐ the claims, Nos.
   - ☐ the drawings, sheets/figs
   - ☐ the sequence listing *(specify)*:
   - ☐ any table(s) related to sequence listing *(specify)*:

4. ☐ This report has been established as if (some of) the amendments annexed to this report and listed below had not been made, since they have been considered to go beyond the disclosure as filed, as indicated in the Supplemental Box (Rule 70.2(c)).
   - ☐ the description, pages
   - ☐ the claims, Nos.
   - ☐ the drawings, sheets/figs
   - ☐ the sequence listing *(specify)*:
   - ☐ any table(s) related to sequence listing *(specify)*:

* *If item 4 applies, some or all of these sheets may be marked "superseded."*

Box No. V Reasoned statement under Article 35(2) with regard to novelty, inventive step or industrial applicability; citations and explanations supporting such statement

1. Statement

Novelty (N)	Yes:	Claims	1-16
	No:	Claims	
Inventive step (IS)	Yes:	Claims	1-16
	No:	Claims	
Industrial applicability (IA)	Yes:	Claims	1-16
	No:	Claims	

2. Citations and explanations (Rule 70.7):

see separate sheet

Re Item I

Basis of the report

The examination is carried out on the following application documents:

Description, pages:	6, 7	as originally filed,
Drawings, sheets:	1/1	as originally filed.
Description, pages:	1-5, 5A	as filed with letter of 18.08.05, received 19.08.05,
Claims, No:	1-16	as filed with letter of 18.08.05, received 19.08.05.

Re Item V

Reasoned statement with regard to novelty, inventive step or industrial applicability; citations and explanations supporting such statement

1. Reference is made to the following document:

   D1: US-A-5569154

2. The following is stated.

2.1 The document D1 discloses (the references in parentheses applying to this document) a device for removing mercury from mercury-containing residues (see column 1, lines 7-11), comprising a gastight (see column 5, lines 20-22) screw conveyor (22) provided with an inlet (12) for mercury-containing residues, first heating means (34) for heating admitted mercury-containing residues and causing mercury to evaporate (see column 6, lines 10-20), an outlet conduit (46) for mercury vapour-containing gas (see column 6, lines 21-24) and an outlet channel (see column 5, lines 38-42) for mercury-free residues, the device being provided with pump means (66)

for applying an underpressure in the screw conveyor (22) and discharging mercury vapour-containing gas therefrom (see column 6, lines 21-24), and with a distillation column (86) provided with cooling means (see column 7, lines 18-22) to cause condensation (see column 6, lines 25-26 and column 7, lines 16-29) of mercury vapour from the mercury vapour-containing gas discharged with the pump means (66).

The subject-matter of claim **1** therefore differs from this known device in that the outlet conduit is provided with second heating means for heating the mercury vapour-containing gas.

The subject-matter of claim **1** is therefore novel and claim **1** meets the requirements of Article 33(2) PCT.

The problem underlying claim **1** is to prevent too strong a cooling of the mercury-containing gas outside the screw conveyor, cf. page 3, line 36 - page 4, line 2.

The solution in accordance with claim **1** is the outlet being provided with second heating means for heating the mercury vapour-containing gas.

The combination of the further features of claim **1** is not disclosed by any of the cited prior art documents.

The skilled person is not hinted at providing the outlet conduit with second heating means for heating the mercury vapour-containing gas in order to solve the problem underlying claim **1**.

The subject-matter of claim **1** thus involves an inventive step and claim **1** meets the requirements of Article 33(3) PCT.

2.2 Under reference to point 2.1 above, it is stated that the document D1 discloses (the references in parentheses applying to this document) a method for removing mercury from mercury-containing residues (see column 1, lines 7-11) with a device as claimed in claim 1, comprising the steps of

(I) admitting mercury-containing residues into a gastight screw conveyor (22) (see column 5, lines 54-57),

(ii) heating the admitted mercury-containing residues and causing mercury to evaporate (see column 6, lines 10-20),

(iii) applying an underpressure in the screw conveyor (22) and discharging mercury vapour-containing gas therefrom (see column 6, lines 21-24),

(iv) causing mercury to condense from the mercury vapour-containing gas discharged with the pump means (66) (see column 6, lines 25-26 and column 7, lines 16-29), and

(v) collecting mercury in a condensed state (see column 7, lines 25-26).

The subject-matter of claim **11** therefore differs from this known method in heating the discharged mercury vapour-containing gas.

The subject-matter of claim **11** is therefore novel and claim **11** meets the requirements of Article 33(2) PCT.

The problem underlying claim **11** is to prevent too strong a cooling of the mercury-containing gas outside the screw conveyor, cf. page 3, line 36 - page 4, line 2.

The solution in accordance with claim **11** is heating the discharged mercury vapour-containing gas.

The combination of the further features of claim **11** is not disclosed by any of the cited prior art documents.

The skilled person is not hinted at heating the discharged mercury vapour-containing gas in order to solve the problem underlying claim **11**.

The subject-matter of claim **11** thus involves an inventive step and claim **11** meets the requirements of Article 33(3) PCT.

3. Claims **2-10** and **12-16** are all truly dependent claims and also meet the requirements of Article 33(1) to 33(5) PCT.

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DG 1
19 08 2005
(44)

DEVICE AND METHOD FOR REMOVING MERCURY FROM RESIDUES

The invention relates to a device for removing mercury from mercury-containing residues, comprising a gastight screw conveyor provided with an inlet for mercury-containing residues, first heating means for heating admitted mercury-containing residues and causing mercury to evaporate, an outlet conduit for mercury vapour-containing gas and an outlet channel for mercury-free residues, the device being provided with pump means for applying an underpressure in the screw conveyor and discharging mercury vapour-containing gas therefrom, and with a distillation column provided with cooling means to cause condensation of mercury vapour from the mercury vapour-containing gas discharged with the pump means. The mercury-containing residues come for instance from luminescence tubes, catalyst material, batteries or drilling sludge.

Such a device is known from the Netherlands patent application no. 9100119.

In the known device about 700 m^3 of ambient air per hour at a temperature of 20°C is heated in an air heater to a temperature of about 300°C and then fed to a heatable screw conveyor into which mercury-containing residues are admitted. Mercury vapour-containing gas having a temperature of about 250°C is discharged via a vapour discharge and admixed to about 2000 m^3 of air with a temperature of 20°C and about 60 m^3 of heated air with a temperature of about 60°C. The thus resulting air-vapour mixture, which has a temperature of about 80°C, is fed to a dust collector and a filter for collecting mercury. In the filter, which consists of active carbon impregnated with sulphur, the mercury is converted to mercury sulphide.

A number of drawbacks are associated with the device known from NL-A-9100119. The mercury in the

mercury-containing residues is evaporated at a pressure of 1 atm. (1 bar) in the device. This has the result that large quantities of air have to be heated and transported in order to actually evaporate all the mercury that may be present. The availability of a voluminous installation is required for this purpose with a high energy requirement and pumps with a relatively high pumping capacity, resulting in high operating and investment costs.

Another drawback of working with air at a pressure of 1 atm. (1 bar) is that the device is not inherently safe. If leakage were to occur at some point, air containing mercury vapour can escape from the known device, with all the risks this entails for the health of people present in the immediate vicinity.

A further drawback of the known device is that the mercury released from the residues becomes available in the form of mercury sulphide, a mercury-containing residual material which must be disposed of as chemical waste.

US-A-5569154 discloses a method for removal of mercury from soil, wherein the soil has been contaminated with mercury. According to this method, soil is added into one end of an internally fired hollow screw desorber under a vacuum. The soil is moved from the one end of the internally hollow screw desorber to another end of the desorber and the temperature of the soil is increased in the desorber as it moves from the one end of the desorber to the other end, wherein the temperature of the soil is increased. A mercury vapour is produced in the internally fired hollow screw desorber. The soil is removed from the other end of the desorber while the mercury vapour is removed from the internally fired hollow screw desorber, whereafter mercury is recovered from the mercury vapour. In this document, no measure is disclosed as how to prevent too

strong a cooling of the mercury-containing gas outside the screw conveyor.

WO-A-0056474 discloses a method and apparatus for use in removing at least one volatile contaminant from contaminated material by using a rotary vacuum retort during high temperature and vacuum processing, in which apparatus elastomeric pinch valve airlocks are employed to isolate the entire system between the airlocks and a vacuum generator. In this document, no measure is disclosed as how to prevent too strong a cooling of separated volatile contaminants outside the rotary vacuum retort.

It is an object of the invention to provide a relatively compact device for removing waste from residues, the investment and operating costs of which are relatively low.

It is another object to provide an inherently safe device, wherein the hopefully unlikely occurrence of leakage does not result in the escape of gases containing mercury vapour.

It is a further object to provide a device, using which mercury is recovered from mercury-containing residues and is not bonded to chemical waste for disposal.

These objectives are achieved, and other advantages gained, with a device stated in the preamble, wherein according to the invention the outlet conduit is provided with second heating means for heating the mercury vapour-containing gas.

The applying of an underpressure achieves that mercury vapour released as a result of the heating in the screw conveyor is immediately discharged in safe manner to the distillation column, where the mercury vapour condenses and the mercury is collected in liquid state and thus becomes available for reuse, whereas in a device according to the invention too strong a cooling

of the mercury-containing gas outside the screw conveyor is prevented.

In an embodiment the inlet of a device according to the invention comprises an inlet funnel provided with a gastight shut-off valve.

The residues, which are supplied for instance in granular state, are herein poured into the inlet funnel, the shut-off valve of which is opened periodically to admit the residues into the screw conveyor.

The first heating means are preferably adapted to heat the admitted residues to a temperature higher than 350°C, more preferably to a temperature higher than 550°C.

From the vapour pressure table of mercury it can be inferred that the amount of air for heating that is necessary to remove the mercury from contaminated residues which occur in practice and have for instance 0.1% by weight of mercury, at a temperature higher than 350°C is low in relation to a determined weight quantity of contaminated residues. This applies to an even greater extent at a temperature higher than 550°C, this temperature being above the melting range of many mercury-containing alloys.

In yet another embodiment, the outlet channel for mercury-free residues comprises an outlet sluice provided with two gastight shut-off valves.

Such an outlet channel provides the practical advantage that mercury-free heated residues can be supplied continuously by the screw conveyor into a part of the outlet channel lying between the screw conveyor and the first of the two shut-off valves, where these residues are left to lie and can cool until the part in question is wholly filled, whereafter the said quantity of residues is received in the sluice by opening the first shut-off valve, and can there cool for a subsequent period until the sluice must be cleared for a

following quantity of residues.

In a practically advantageous embodiment, the outlet conduit comprises a dust filter for intercepting dust which comes from the residues and which is entrained by the mercury vapour-containing gas.

In order to achieve that the amount of air, even when it cools outside the heated screw conveyor, is sufficient to hold in vapour form the mercury from the residues, and to prevent premature condensation, the device is provided in a preferred embodiment with air inlet means and control means for admitting air for the purpose of receiving therein and discharging mercury vapour, while maintaining the pressure in the device at a predetermined value.

In another preferred embodiment, the pump means are provided with third heating means for heating the mercury vapour-containing gas.

The second and third heating means are for instance adapted to maintain the temperature of the mercury vapour-containing gas at a value of at least 180°C.

In order to ensure that all mercury vapour from the mercury vapour-containing gas fed into the distillation column condenses therein, the cooling means are adapted to cool the mercury vapour-containing gas to a temperature at least lower than minus 30°C.

The invention further relates to a method for removing mercury from mercury-containing residues using the device described here, comprising the steps of (i) admitting mercury-containing residues into a gastight screw conveyor, (ii) heating the admitted mercury-containing residues and causing mercury to evaporate, (iii) applying an underpressure in the screw conveyor and discharging and heating mercury vapour-containing gas therefrom, (iv) causing mercury vapour to condense from the mercury vapour-containing gas discharged with

the pump means, and (v) collecting mercury in condensed state.

The invention will be elucidated hereinbelow on the basis of an exemplary embodiment and with reference to the drawing.

In the drawing fig. 1 shows a simplified block diagram of an exemplary embodiment of a device 1 according to the invention, with a screw conveyor 2 which is provided with a gastight casing 3 in which heating elements (not shown) are arranged, and which is driven by a motor 4. Screw conveyor 2 is disposed at an angle of 30° to a horizontal plane, is provided on its inlet side (on the left in the figure) with an inlet funnel 5 for granular residues, or at least residues reduced in size, (represented by arrow 6) and is provided on its outlet side (on the right in the figure) with an outlet conduit 7 for mercury vapour-containing gas, with dust filter 8, and an outlet channel 10 for mercury-free residues (represented by arrow 11). Inlet funnel 5 is provided with a gastight shut-off valve 9, outlet channel 10 is provided with two gastight shut-off valves 12, 13, between which a sluice 14 is formed, and debouches above a collecting bin 15. A vacuum pump 16 is connected on its inlet side to outlet conduit 7 and connected on its outlet side to a distillation column 17, which is provided with a Vigreux cooler 18 for pre-cooling the mercury vapour-containing gas to a temperature of about 20°C, and with an intensive cooler 19 for further cooling to a temperature of minus 38°C. A collecting vessel 20 is provided for collecting condensed (liquid) mercury which slides downward along the walls of distillation column 17. In order to prevent mercury condensing prematurely in outlet conduit 7 or pump 16, both of these are provided with heating elements (not shown). In order to maintain the pressure in screw conveyor 2 at a predetermined value in

CLAIMS

1. Device (1) for removing mercury from mercury-containing residues (6), comprising a gastight screw conveyor (2) provided with an inlet (5) for mercury-containing residues (6), first heating means for heating admitted mercury-containing residues and causing mercury to evaporate, an outlet conduit (7) for mercury vapour-containing gas and an outlet channel (10) for mercury-free residues, the device (1) being provided with pump means (16) for applying an underpressure in the screw conveyor (2) and discharging mercury vapour-containing gas therefrom, and with a distillation column (17) provided with cooling means (18, 19) to cause condensation of mercury vapour from the mercury vapour-containing gas discharged with the pump means (16), characterized in that the outlet conduit (7) is provided with second heating means for heating the mercury vapour-containing gas.

2. Device (1) as claimed in claim 1, characterized in that the inlet comprises an inlet funnel (5) provided with a gastight shut-off valve (9).

3. Device (1) as claimed in claims 1-2, characterized in that the first heating means are adapted to heat the admitted residues to a temperature higher than 350°C.

4. Device (1) as claimed in claim 3, characterized in that the first heating means are adapted to heat the admitted residues to a temperature higher than 550°C.

5. Device (1) as claimed in any of the claims 1-4, characterized in that the outlet channel (10) for mercury-free residues comprises an outlet sluice (14) provided with two gastight shut-off valves (12,13).

6. Device (1) as claimed in any of the claims 1-5, characterized in that the outlet conduit (7) comprises a dust filter (8).

7. Device (1) as claimed in any of the claims 1-6, characterized in that it is provided with air inlet means (21) and control means for admitting air for the purpose of receiving therein and discharging mercury vapour.

8. Device (1) as claimed in any of the claims 1-7, characterized in that the pump means (16) are provided with third heating means for heating the mercury vapour-containing gas.

9. Device (1) as claimed in claims 7 and 8, characterized in that the second and third heating means are adapted to maintain the temperature of the mercury vapour-containing gas at a value of at least 180°C.

10. Device (1) as claimed in any of the claims 1-9, characterized in that the cooling means (19) are adapted to cool the mercury vapour-containing gas to a temperature at least lower than minus 30°C.

11. Method for removing mercury from mercury-containing residues (6) with a device (1) as claimed in claim 1, comprising the steps of

(i) admitting mercury-containing residues (6) into a gastight screw conveyor (2),

(ii) heating the admitted mercury-containing residues and causing mercury to evaporate,

(iii) applying an underpressure in the screw conveyor (2) and discharging and heating mercury vapour-containing gas therefrom,

(iv) causing mercury vapour to condense from the mercury vapour-containing gas discharged with the pump means (16), and

(v) collecting mercury in condensed state.

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12. Method as claimed in claim 11, characterized in that the residues are heated in step (ii) to a temperature of 560°C.

13. Method as claimed in any of the claims 11-12, characterized in that the underpressure to be applied in step (iii) amounts to 750 mbar.

14. Method as claimed in any of the claims 11-13, characterized in that the mercury vapour-containing gas to be discharged in step (iii) is guided into a heated conduit (7) in which this gas is held at a temperature of at least 180°C.

15. Method as claimed in any of the claims 11-14, characterized in that the mercury vapour-containing gas to be discharged in step (iv) is guided into a distillation column (17) which is at least partially cooled to a temperature of minus 38°C.

16. Method as claimed in any of the claims 11-15, characterized in that while maintaining the underpressure to be applied in step (iii) air is admitted into the device (1) in order to entrain mercury vapour.